The conformations of (1) and (2) differ significantly owing to the differences in ring fusion. The five-membered ring in both compounds is in an envelope conformation; however, C(4) is the flap in (1) while C(1) is the flap in (2). The distances and angles around the ring cannot be compared owing to differences in functionality. The seven-membered heptene ring in (2) has an almost perfect chair conformation while that in (1) is a highly distorted twist chair (Toromanoff, 1980; Bucourt, 1974), see torsion angles in Table 3. The C(1)—C(5) bond common to both rings differs significantly, 1·578 (5) Å for (1) and 1·552 (6) Å for (2), as do a number of valence angles. The ester groups are not held tightly by packing interactions and the thermal parameters are relatively large as are the thermal parameters for the C(12) and C(13) methyl groups in (2).

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Structural Studies of Pyrimidine Cyclonucleoside Derivatives. IX.* Structure of 6,2′-Anhydro-6-ethyl-1-β-D-arabinofuranosyluracil

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Abstract. $C_{11}H_{14}N_2O_5$, $M_r = 254{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}400\,(1)$, $b = 9{\cdot}734\,(2)$, $c = 17{\cdot}797\,(5)$ Å, $V = 1108{\cdot}7\,(4)$ Å^3, $Z = 4$, $D_m = 1{\cdot}506\,(1)$, $D_x = 1{\cdot}523$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}69$ mm^{-1}, $F(000) = 536$, $T = 290$ K, $R = 0{\cdot}057$ for 1030 independent observed reflections. The glycosidic torsion angle is in the *anti* range, $\chi_{\rm CN}$ [O(4′)—C(1′)—N(1)—C(2)] = 268·1 (3)°, and the sugar conformation is the unusual $^{\rm O}T_4$. The O atom O(5′) takes two disordered orientations, *gauche–trans* and *trans–gauche*, but not *gauche–gauche* (*gauche* ≡ −synclinal) as frequently found in nucleosides. No base stacking is observed.

Introduction. Cyclonucleosides have generally served as good model compounds for investigations of physicochemical properties of nucleosides and nucleotides in solution because they have conformational rigidity between the base and the sugar moieties by cyclization (Rogers & Ulbricht, 1970; Ikehara, Kaneko, Nakahara, Yamada & Uesugi, 1971; Ueda & Shibuya, 1974; Manor, Saenger, Davies, Jankowski & Rabczenko, 1974; Yamagata, Fujii, Fujiwara, Tomita & Ueda, 1981). In particular, cyclonucleosides cyclized through a C atom are much better model nucleosides for spectroscopic studies because the bridging C atom has less effect on the physicochemical properties of the base or the sugar moiety than O or S bridges, and we recently synthesized several new types of C-bridged

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* Part VIII: Yamagata, Fujiwara, Tomita, Shuto & Ueda (1984).

pyrimidine cyclonucleosides and measured their CD spectra (Ueda, Shuto, Sano, Usui & Inoue, 1982; Ueda, Usui, Shuto & Inoue, 1984; Ueda & Shuto, 1984). In order to determine the glycosidic torsion angles and overall conformations of the cyclonucleosides and to investigate the relationships between their conformations and CD patterns, we have carried out the structure analysis of the title compound, 6,2′-C_2-cyclo-U, which is the first example of a C-bridged pyrimidine cyclonucleoside determined by the X-ray diffraction method. This compound possesses a six-membered ring by cyclization instead of a five-membered ring as already found in 2,2′- and 6,2′-cyclonucleosides (Delbaere & James, 1973; Suck & Saenger, 1973; Yamagata, Fujii, Kanai, Ogawa & Tomita, 1979; Yamagata, Koshibe, Tokuoka, Fujii, Fujiwara, Kanai & Tomita, 1979; Yamagata, Suzuki, Fujii, Fujiwara & Tomita, 1979; Yamagata, Yoshimura, Fujii, Fujiwara, Tomita & Ueda, 1980).

Experimental. Title compound synthesized as described in a previous paper (Ueda *et al.*, 1982) and crystallized from ethanol solution as colorless prisms; density by flotation in C_6H_6–CCl_4 mixture; crystal 0·2 × 0·25 × 0·4 mm, Rigaku automatic four-circle diffractometer, ω–2θ scanning technique, $2\theta_{\max} = 125°$, graphite-monochromatized Cu $K\alpha$ radiation; unit-cell dimensions by least-squares procedure based on 2θ values ($39 < 2\theta < 60°$) of 18 reflections; intensity fluctuation monitored periodically by three reflections (200, 080, 0,0,12): <4%; 1061 independent reflections ($0 \le h \le 7$, $0 \le k \le 11$, $0 \le l \le 20$), 1037 observed with $F_o \ne 0$; Lorentz and polarization corrections; direct methods (*MULTAN*78, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); full-matrix least-squares method with anisotropic temperature factors for all non-H atoms; all H atoms except those attached to O(5′) and C(5′) located on difference Fourier map; the population of disordered O(5′) atom determined from peak height in the Fourier map excluding O(5′) atom, 0·6:0·4; final refinement including H atoms with isotropic temperature factors reduced R to 0·057 ($wR = 0\cdot080$, $S = 1\cdot12$)[*] for 1030 reflections; seven low-angle reflections (031, 020, 021, 022, 011, 101, 002) omitted from final refinement for their extinction effects; function minimized $\sum w(|F_o| - k|F_c|)^2$, where $w = 1/[\sigma^2(F_o) + 0\cdot017|F_o| + 0\cdot0036|F_o|^2]$ and k is the scale factor; largest peaks in final difference Fourier map: 0·22 e Å^{-3}; $(\Delta/\sigma)_{\rm av} = 0\cdot03$ and $(\Delta/\sigma)_{\max} = 0\cdot13$, for non-H atoms; scattering factors from *International Tables for Crystallography* (1974); program system *UNICS* (1979) on an ACOS 900 computer of the Crystallographic Research Center, Institute for Protein Research, Osaka University.

Discussion. Tables 1 and 2 give the final atomic parameters and the bond distances and angles, respectively. Fig. 1 is an *ORTEP*II drawing (Johnson, 1976) of 6,2′-C_2-cyclo-U, and Fig. 2 shows the molecular packing with the hydrogen-bonding scheme.

Table 1. *Final atomic coordinates of non-*H *atoms and equivalent isotropic thermal parameters with their e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
N(1)	−0·0488 (4)	0·4521 (3)	0·8116 (1)	1·9 (1)
C(2)	−0·1936 (6)	0·4723 (4)	0·8688 (2)	2·5 (1)
N(3)	−0·1313 (5)	0·4345 (4)	0·9381 (2)	2·7 (1)
C(4)	0·0653 (6)	0·3819 (4)	0·9579 (2)	2·5 (1)
C(5)	0·2016 (6)	0·3587 (4)	0·8955 (2)	2·3 (1)
C(6)	0·1444 (6)	0·3928 (3)	0·8252 (2)	1·9 (1)
O(2)	−0·3645 (4)	0·5227 (4)	0·8573 (2)	4·0 (1)
O(4)	0·1065 (5)	0·3604 (3)	1·0240 (1)	3·3 (1)
C(1′)	−0·1139 (5)	0·4841 (4)	0·7360 (2)	2·0 (1)
C(2′)	0·0576 (5)	0·5364 (3)	0·6829 (2)	1·9 (1)
C(3′)	0·0080 (5)	0·4613 (4)	0·6092 (2)	1·8 (1)
C(4′)	−0·1970 (5)	0·3870 (4)	0·6243 (2)	2·1 (1)
C(5′)	−0·2252 (10)	0·2512 (5)	0·5854 (3)	4·5 (1)
C(6′)	0·2808 (5)	0·3744 (4)	0·7575 (2)	2·3 (1)
C(7′)	0·2774 (5)	0·5083 (4)	0·7127 (2)	2·4 (1)
O(4′)	−0·1962 (4)	0·3623 (3)	0·7034 (1)	2·3 (1)
O(3′)	−0·0197 (4)	0·5520 (3)	0·5474 (1)	3·0 (1)
O(5′A)	−0·2390 (14)	0·2535 (11)	0·5123 (3)	6·8 (2)
O(5′B)	−0·4489 (16)	0·2267 (9)	0·5794 (6)	5·6 (3)

Table 2. *Bond distances* (Å) *and angles* (°) *for the non-*H *atoms*

N(1)–C(2)	1·391 (5)	C(1′)–C(2′)	1·535 (5)
N(1)–C(6)	1·386 (4)	C(2′)–C(3′)	1·534 (5)
C(2)–N(3)	1·348 (5)	C(3′)–C(4′)	1·522 (5)
N(3)–C(4)	1·403 (5)	C(4′)–O(4′)	1·427 (4)
C(4)–C(5)	1·430 (5)	C(4′)–C(5′)	1·503 (6)
C(5)–C(6)	1·344 (5)	C(2′)–C(7′)	1·528 (5)
N(1)–C(1′)	1·443 (4)	C(7′)–C(6′)	1·528 (5)
C(2)–O(2)	1·216 (5)	C(3′)–O(3′)	1·421 (4)
C(4)–O(4)	1·224 (5)	C(5′)–O(5′A)	1·30 (1)
C(6)–C(6′)	1·499 (5)	C(5′)–O(5′B)	1·46 (1)
C(1′)–O(4′)	1·422 (4)		
C(2)–N(1)–C(6)	121·7 (3)	O(4′)–C(1′)–C(2′)	106·9 (3)
C(2)–N(1)–C(1′)	117·4 (3)	C(1′)–C(2′)–C(3′)	102·7 (3)
C(6)–N(1)–C(1′)	120·8 (3)	C(1′)–C(2′)–C(7′)	112·6 (3)
N(1)–C(2)–N(3)	115·8 (3)	C(3′)–C(2′)–C(7′)	113·7 (3)
N(1)–C(2)–O(2)	122·0 (3)	C(2′)–C(3′)–C(4′)	104·7 (3)
N(3)–C(2)–O(2)	122·3 (3)	C(2′)–C(3′)–O(3′)	113·0 (3)
C(2)–N(3)–C(4)	126·4 (3)	C(4′)–C(3′)–O(3′)	108·9 (3)
N(3)–C(4)–C(5)	114·2 (3)	C(3′)–C(4′)–O(4′)	104·6 (3)
N(3)–C(4)–O(4)	119·8 (3)	C(3′)–C(4′)–C(5′)	116·1 (3)
C(5)–C(4)–O(4)	126·1 (3)	O(4′)–C(4′)–C(5′)	107·8 (3)
C(4)–C(5)–C(6)	121·2 (3)	C(1′)–O(4′)–C(4′)	105·2 (2)
N(1)–C(6)–C(5)	120·6 (3)	C(4′)–C(5′)–O(5′A)	116·9 (5)
N(1)–C(6)–C(6′)	115·3 (3)	C(4′)–C(5′)–O(5′B)	107·2 (5)
C(5)–C(6)–C(6′)	124·1 (3)	C(2′)–C(7′)–C(6′)	110·3 (3)
N(1)–C(1′)–O(4′)	107·9 (3)	C(7′)–C(6′)–C(6)	108·0 (3)
N(1)–C(1′)–C(2′)	116·1 (3)		

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[*] Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and selected torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42367 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The glycosidic torsion angle, χ_{CN}, is 268·1 (3)°, which corresponds to an *anti* conformation and is comparable with that (272·7°) of 7,2′-anhydro-β-D-arabinofuranosylorotidine (7,2′-*O*-cyclo-O; Smith, Chwang & Sundaralingam, 1980) having the newly formed similar six-membered fused ring but is apparently different from that (291·4°) of 6,2′-anhydro-1-β-D-arabinofuranosyl-6-hydroxycytosine (6,2′-*O*-cyclo-C; Yamagata, Fujii, Kanai, Ogawa & Tomita, 1979) with the five-membered fused ring. This difference is caused by the variety of conformations in each cyclic ring system between the base and the sugar, *i.e.* 6,2′-*O*-cyclo-C forms a rigid planar fused ring by cyclization but the six-membered fused ring formed by 7,2′-*O*-cyclo-O or 6,2′-C_2-cyclo-U adopts the more flexible envelope conformation with displacement of the C(2′) atom from the plane formed by the remaining atoms or with displacements of C(2′) and C(7′) atoms, respectively. In this molecule, the sugar conformation is the unusual $^{O}T_4$ ($P = 81{\cdot}5°$, $\psi_m = 40{\cdot}7°$). The conformations of the fused rings depend on the conformations of the sugar rings as well as the glycosidic conformations. The planar conformation of the fused ring in 6,2′-*O*-cyclo-C and the envelope conformation in 6,2′-C_2-cyclo-U and 7,2′-*O*-cyclo-O result in a variation of the endocyclic C(1′)—C(2′) torsion angle [6·2, 18·2 (3) and 41·0°], and also of the sugar puckering [E_4 ($P = 64°$, $\psi_m = 45°$), $^{O}T_4$ ($P = 81{\cdot}5°$, $\psi_m = 40{\cdot}7°$) and $^{2}T_1$ ($P = 132{\cdot}1°$, $\psi_m = 41{\cdot}4°$)]. All these conformations are favorable for preventing short contacts between the C(5′) atom and the fused-ring moiety. Two disordered orientations of the O(5′) atom are *gauche–trans* and *trans–gauche*, but are not *gauche–gauche* as frequently found in nucleosides.

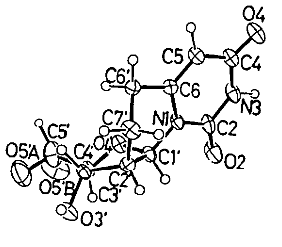

Fig. 1. Molecular conformation and atom numbering.

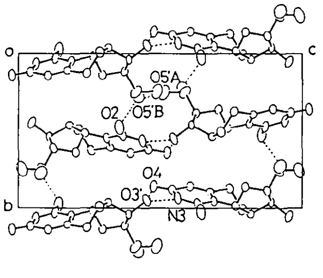

Fig. 2. Molecular packing viewed down the *a* axis. Dotted lines indicate the hydrogen bonds.

The bond distances and angles are quite normal in comparison with standard nucleosides (Taylor & Kennard, 1982; Sundaralingam, 1973), but differ from those of the five-membered 2′-cyclonucleosides with the strain around the C(1′) and C(2′) atoms (Suck & Saenger, 1973; Delbaere & James, 1973; Yamagata, Fujii, Kanai, Ogawa & Tomita, 1979; Yamagata, Koshibe, Tokuoka, Fujii, Fujiwara, Kanai & Tomita, 1979; Yamagata, Suzuki, Fujii, Fujiwara & Tomita, 1979; Yamagata, Yoshimura, Fujii, Fujiwara, Tomita & Ueda, 1980).

In the present crystal no base stacking is observed. There are four kinds of hydrogen bonds, *i.e.* N(3)—H···O(3′) [2·965 (4) Å], O(3′)—H···O(4) [2·810 (4) Å], O(5′*B*)—H···O(2) [2·58 (1) Å] and O(5′*A*)—H···O(5′*B*) [2·48 (1) Å] as shown in Fig. 2. Of these the formation of the O(5′*A*)—H···O(5′*B*) hydrogen bond is ambiguous because of the disorder of both atoms.

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Structural Studies of Pyrimidine Cyclonucleoside Derivatives. X.* Structure of 5'-*O*-Acetyl-6,3'-anhydro-2'-deoxy-6-methyl-1-β-D-xylofuranosyluracil

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Abstract. $C_{12}H_{14}N_2O_5$, $M_r = 266{\cdot}3$, monoclinic, $P2_1$, $a = 8{\cdot}312\,(1)$, $b = 5{\cdot}559\,(1)$, $c = 13{\cdot}048\,(2)$ Å, $\beta = 98{\cdot}50\,(2)^\circ$, $V = 596{\cdot}2\,(2)$ Å^3, $Z = 2$, $D_m = 1{\cdot}480\,(1)$, $D_x = 1{\cdot}483$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}68$ mm^{-1}, $F(000) = 280$, $T = 293$ K, $R = 0{\cdot}049$ for 1055 independent observed reflections. The glycosidic torsion angle, $\chi_{\mathrm{CN}}[\mathrm{O}(4')\text{–}\mathrm{C}(1')\text{–}\mathrm{N}(1)\text{–}\mathrm{C}(2)] = 265{\cdot}3\,(5)^\circ$, is in the *anti* range, and the furanose conformation is E_2. The orientation of O(5') is *trans–gauche* (*gauche* ≡ –synclinal). No base stacking is observed. The molecules form infinite columns along the *b* axis by N(3)—H···O(4) hydrogen bonds between adjacent molecules related by twofold screw symmetry.

Introduction. As described in the preceding paper (Yamagata, Okabe, Tomita, Shuto, Inoue & Ueda, 1985), C-bridged cyclonucleosides provide important information about the relationship between glycosidic torsion angles and CD spectra (Ueda, Shuto, Sano, Usui & Inoue, 1982; Ueda; Usui, Shuto & Inoue, 1984; Ueda & Shuto, 1984). In this paper we deal with the structure determination of the title compound abbreviated as 6,3'-*C*-cyclo-U, as the second member of a series of C-bridged pyrimidine cyclonucleosides. 6,3'-*C*-Cyclo-U is the first example of a pyrimidine cyclonucleoside with a covalent bond between C(3') in the sugar ring and C(6) in the normal pyrimidine base determined by X-ray diffraction.

Experimental. Title compound synthesized as described in a previous paper (Ueda & Shuto, 1984) and crystallized from methanol solution as colorless prisms; density by flotation in C_6H_6–CCl_4 mixture; crystal $0{\cdot}15 \times 0{\cdot}25 \times 0{\cdot}6$ mm, Rigaku automatic four-circle diffractometer, ω–2θ scanning technique, $2\theta_{\max} = 125^\circ$, graphite-monochromatized Cu $K\alpha$ radiation; unit-cell dimensions by least-squares procedure based on 2θ values ($20 < 2\theta < 47^\circ$) of 24 reflections; intensity fluctuation monitored periodically by three reflections ($\bar{5}00$, 020, 008): <2%; 1061 independent reflections ($-9 \le h \le 9$, $0 \le k \le 6$, $0 \le l \le 14$), 1055 observed with $F_o \ne 0$; Lorentz and polarization corrections; structure solved by direct interpretation of Patterson function: peaks around origin indicated orientation of uracil and cyclic six-membered rings, atomic positions determined by packing consideration; full-matrix least-squares method with anisotropic temperature factors for all non-H atoms; all H atoms located on difference Fourier map; final refinement including H atoms with isotropic temperature factors reduced R to $0{\cdot}049$ ($wR = 0{\cdot}053$, $S = 7{\cdot}59$);† function minimized $\sum w(|F_o| - k|F_c|)^2$, where $w = 1/\sigma^2(F_o)$ and k is the scale factor; largest peaks in final difference Fourier map: $0{\cdot}22$ e Å^{-3}; $(\Delta/\sigma)_{\mathrm{av}} = 0{\cdot}02$ and $(\Delta/\sigma)_{\max} = 0{\cdot}08$, for non-H atoms; scattering factors from *International Tables for X-ray Crystallography* (1974); program system *UNICS* (1979) on an ACOS 900 computer of the Crystallographic Research Center, Institute for Protein Research, Osaka University.

^{1}H NMR spectra in $CDCl_3$ solution measured by a JEOL FX-200FT NMR spectrometer with Me_4Si as an internal reference.

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* Part IX: Yamagata, Okabe, Tomita, Shuto, Inoue & Ueda (1985).

† Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and selected torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42368 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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